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M.G. Kamath, G.S. Bhat, D.V. Parikh and B.D. Condon

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Processing and Characterization of Flame Retardant Cotton Blend Nonwovens for Soft Furnishings to Meet Federal Flammability Standards

M. G. KAMATH AND G. S. BHAT*
The University of Tennessee, Knoxville, TN, USA

D. V. PARIKH AND B. D. CONDON
SRRC-ARS-USDA, New Orleans, LA, USA

ABSTRACT: Effective from July 1, 2007 it is mandatory that all mattress sets meet the federal flammability standard CFR 1633. It is necessary to impart flame resistance that would provide at least 30 min for occupants to escape fire. Changes in the flammability laws are expected on other soft furnishings of sleep products like comforters and pillows. Generally these products are often the first to be engulfed by the fire. Currently many inherently flame retardant (FR) fibers and chemicals are available in the market. We have developed barrier fabrics with FR properties by incorporating these fibers in blends with cotton that either meet or exceed the standard. Results from this ongoing research are discussed in this article.

KEY WORDS: cotton, CFR 1633, flammability, flame retardant, FR, soft furnishings, sleep products, comforters, nonwovens, pillows, mattress, bedding, fibers, barrier, LOI, TB603, and TB604.

INTRODUCTION

RECENTLY, THE SCIENTIFIC research on bedding products is focusing on the flame retardancy regulations enforced by the legislature.

*Author to whom correspondence should be addressed.
E-mail: gbhat@utk.edu
Figures 1 and 5–7 appear in color online: http://jit.sagepub.com

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These regulations are formulated time to time based on the data provided by the U.S. Consumer Product Safety Commission (CPSC) on residential fires [1]. National Cotton Batting Institute also provides relevant information on these products [2]. Major concern is the open flame test, 16 CFR Part 1633 effective since July 1, 2007. The primary objective of flame-retardants is to save lives by providing more time for one to escape from the fire. Hence all bedding manufacturers are looking for FR treatment of their products to meet these standards. This highlights the vast market potential for a viable FR barrier product with improved flame retardancy to meet the flammability regulations.

Cotton is comfortable, a natural product, a renewable resource, and environmentally friendly. Cotton-based nonwoven battings [3] have been used in bedding products for years. The early mattress [4] was just a bag filled with the natural fibers such as straw, horsehair, wool, coconut fiber, or cotton. Today, a typical mattress in the United States is constructed of several layers of foam, natural and synthetic fibers, nonwovens, and springs. The main purpose of the mattress [5] is to provide adequate sleep comfort. Research [6] indicates that a mattress that conforms better to the shape of the body provides better support and comfort. Also, natural fibers such as cotton and wool can take perspiration away from the skin and keep one cooler and comfortable.

Materials such as stone, brick, concrete, and glass are intrinsically flame resistant that simply do not burn. As early as 1735, Obadiah Wyld developed a mixture of chemicals that imparts FR properties. Boric acid treatment of cotton was developed to meet standard cigarette ignition resistance, and imparts a certain level of flame resistance, but is inadequate to meet open flame standard. Performance limitations [7] are determined by stability of the chars produced following interaction of the FR chemicals and the fiber when heated. Pretreated cellulosic FR fibers are available at reasonably low cost. Furthermore, there are synthetic fibers such as aramids, novoloids, polyamide imides, carbonized acrylics, and melamines that are inherently flame resistant [8], but these fibers are very expensive, and hence introduce a spike in the cost of the product. Moreover, jumps in crude oil prices will have a direct impact on all these synthetic fibers unlike cotton, which is less sensitive to changes in the crude oil price.

Flame retardants function by interfering in at least one of the three components of combustion heat, fuel, and oxygen. In the case of melamine fibers, it functions as a heat sink at the early stages, then it degrades to evolve nitrogen that dilutes the vapor and in the later stage the residue acts as a barrier between air and fuel. There are a few chemicals such as bromides, that interfere in the burning reaction and stop the spread of flames, but their combustion products are toxic. Metal hydroxides are safe, quench the fire by releasing cooling gases, but loading ATH into the fiber to the desired level ($\sim$50%) is not feasible. Phosphorus compounds are good candidates to incorporate into a polyester fiber (loading to a desired extent $\sim$10%) and act as char-forming agents when exposed to the flame. There are chemicals to provide intumescence effect, a foamy barrier that prevents further decomposition of the fuel.

Generally bedding products use high-loft nonwovens made up of cotton and other fibers. Pretreated cotton is blended with a tailored mixture of FR fibers. Earlier studies [9] showed that improved FR and physical resiliency could be obtained from a single-bath chemical finishing using diammonium phosphate (DAP), along with the crosslinking agent dimethylol dihydroxyethylene urea (DMDHEU). Various FR chemicals [10], FR finishes [11], and microencapsulating [12] techniques are found in commercial applications. Feedback processes [13] during thermal decomposition of treated cotton, effect of various phosphorus [14] compositions and mechanisms [15] involved have been explained. Enhancement of FR along with antibacterial properties has been tried [16]. Rhoplex [17] is a binder chemical available in the market to adhere FR chemicals onto fibers. Incorporation of FR flexible foam or a Spunfab [18] dry adhesive in a sandwich pattern is another method to impart FR properties.

Our earlier experience [19] proves that an intimate blend is the key to success. Carding is a well-known process for making an intimate blend of fibers. Through-air thermo-bonding method integrates the web as well as cures the adhesive resulting in strong highloft nonwovens. This research focused on developing a FR highloft nonwoven using feedstock consisting of cotton and other FR fibers and FR chemicals that are commercially available in sufficient quantities at a reasonably low price. Thus, the product of this research would be tailored precisely for sleep products (such as pillows, mattress pads, or barrier material in mattress sets) such that they conform to the latest open flame standard.

EXPERIMENTAL DETAILS

In this research, mechanically cleaned unbleached gray cotton and other commercial grade FR fibers obtained from various industries and organizations were used. Experiments were carried out to produce nonwoven webs using these fibers, with cotton as the major component fiber. These fibers were mixed in the desired proportion using a carding machine (SDS Atlas) to prepare a uniform blend of fibers ($\sim$300 mm wide webs), which is expected to be uniform throughout the product.

Table 1. Details of fiber blend.

	Control	Combination I	Combination II
% Binder fiber	15	15	15
% Cotton	85	50	50
% Cellulosic FR	0	20	20
% Synthetic FR 1*	0	15	0
% Synthetic FR 2*	0	0	15

Note: The identity of FR chemicals and FR fibers are not disclosed because of the intention for filing a patent.

Table 2. Details of FR chemical treatment.

	Control	Set 1	Set 2	Set 3	Set 4
% FR Chemical 1*	0	5	10	15	20
% FR Chemical 2*	0	5	10	15	20

Note: The identity of FR chemicals and FR fibers are not disclosed because of the intention for filing a patent.

Binder fibers are blended to assist in through air bonding. These binder fibers have the low melting (~80°C) polyester sheath with the regular polyester core melting at ~250°C. The process conditions for through air bonding were optimized to impart sufficient strength for further handling, loftiness, and appearance. Table 1 shows the various combinations of fibers used to produce the webs.

The carded webs with a basis weight of ~300 g/m^2 and a thickness of ~15mm were prepared and used in all experiments. FR chemicals such as boric acid, phosphates, silicates, and nanoclays are applied to the blended fiber web as a solution or slurry (~20% concentration) in the presence of a necessary dispersing agent, and bonding or crosslinking agent using the Mathis Laboratory Equipment (Two Roll Padder Type VFM and Oven/Dryer Type KTF-S) for dipping, coating, padding, hot air assisted curing, and drying. The various sets of samples produced are listed in Table 2. The process conditions were optimized to obtain sufficient adherence without appreciable loss in loftiness and appearance (Figure 1).

Other experimental variables are mixtures of FR Chemical 1 & FR Chemical 2, and FR Chemical 3 (containing nanoclay). Proprietary dispersion agents (1% based on the additive) were added while preparing a solution/slurry of chemicals in water (20% concentration). Similarly proprietary-bonding agents (~10% based on the additive) were added and mixed in the slurry just before the application. Calculated amount of solution/slurry was weighed and incorporated into the nonwoven web using

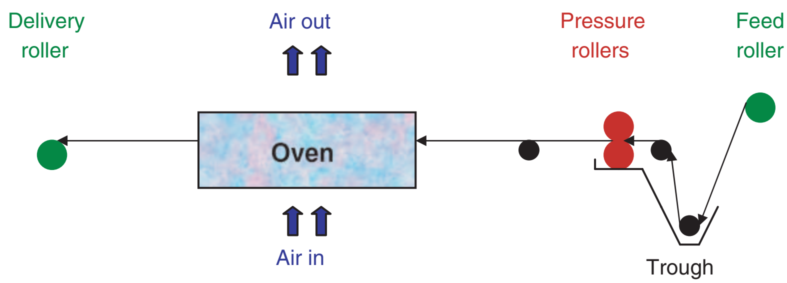

FIGURE 1. Schematic of the Mathis dip squeeze coat and cure equipment.

Mathis equipment through the dip, squeeze (0.5bar pressure), and cure-dry process at 125°C.

The samples produced in the experiments were analyzed after conditioning the samples for at least 24 h under standard laboratory conditions, which are 21°C ± 1°C and 65% ± 10% relative humidity.

Samples were tested for Limiting Oxygen Index (LOI) levels using the General Electric Flammability Tester according to ASTM D2863 method. LOI is the minimum concentration of oxygen that will just support flaming combustion in a flowing mixture of oxygen and nitrogen. A specimen is positioned vertically in a transparent test column and a mixture of oxygen and nitrogen is flown upward through the column. The specimen is ignited at the top with a flame. The oxygen concentration is adjusted until the specimen just supports (flame) combustion. The concentration reported is the volume percent. A 50 mm wide, 150 mm long rectangular specimen was used for the test. LOI tests were repeated five or more times until the results were concordant.

The FR nonwoven webs produced were evaluated for flammability, mechanical properties, morphology, and the nature of adhesion between the chemical and the fiber. After the burn tests the char was observed for the structural integrity and intumescent behavior. The Scanning Electron Microscope (SEM) photographs were obtained using Leo 1525 surface scanning electron microscope in back scatter mode with Gemini column at system vacuum of about 1.3×10^{-5}torr and at an acceleration voltage ~5 kV. SPI Module sputter coater was used to coat the samples with gold for 5 s at 20 mA plasma current to reduce charging while scanning. SEM pictures were taken to understand the morphology. Pictures were taken at the desired magnification focusing on various fibers, fiber-to-fiber bond points, adhered additives, etc. Further, the same sample was analyzed for Energy Dispersive X-ray Spectrum (EDS or EDAX) to identify the elements (Al, P, Si, etc.) present in the sample. The EDS does not detect Boron, since it is outside the detection range.

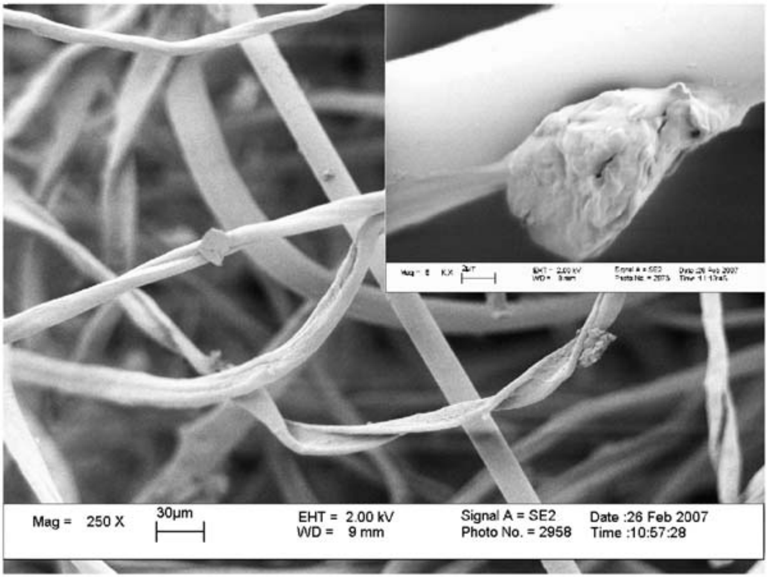

FIGURE 2. SEM picture of FR chemical treated nonwoven.

RESULTS AND DISCUSSION

The cotton-rich nonwoven webs containing 15% binder fibers achieved optimum thermo-bonding at the hot air temperature of 175°C and residence time of 3 min, to impart sufficient strength for further handling. The SEM picture (Figure 2) of FR treated nonwovens clearly shows the presence of constituent fibers and additives adhering onto their surface. Inset picture at 5000 times magnification shows adherence at sub-micron level. There are no loose or unattached additives detected. The SEM picture of the after burn char (Figure 3) shows a reinforcing grid of undestroyed FR fibers supporting the char formed out of cellulosic fibers with the FR additives still present and strengthening the char. Moreover, the char containing FR Chemical 2 exhibited improved structural integrity, and a minor swell of intumescence. At the same time the char containing FR Chemical 1 was fluffy and loose. The EDS picture (Figure 4) confirms presence of Al, P, Si; the constituents of FR chemicals bonded to the nonwoven web.

By blending cotton with FR fibers (35%), the LOI value increases from 19 for cotton to 22. Flame retardancy is further improved by chemical treatment. LOI results (Figure 5) indicate further increase in LOI from 22 to 38 as the chemical loading increased up to 20%. Moreover, the results show superior performance of FR Chemical 2 compared to FR Chemical 1.

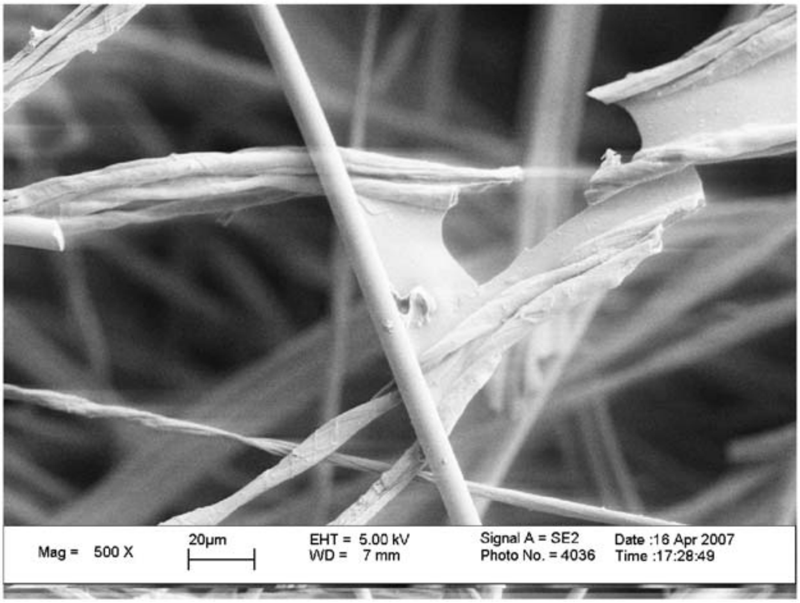

FIGURE 3. SEM picture of after burn char.

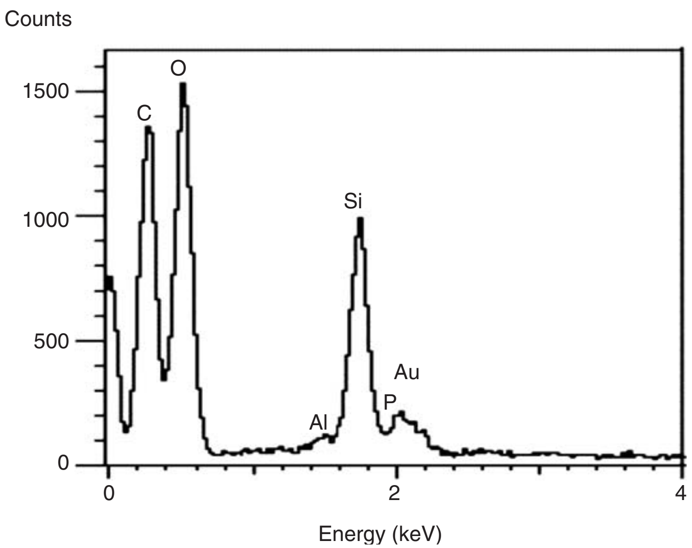

FIGURE 4. EDS Graph of FR chemical treated nonwoven.

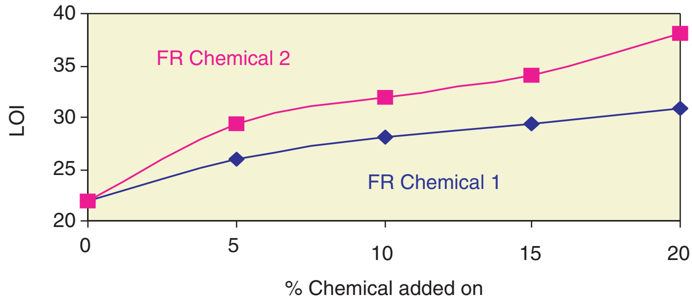

FIGURE 5. LOI of FR chemical treated nonwovens.

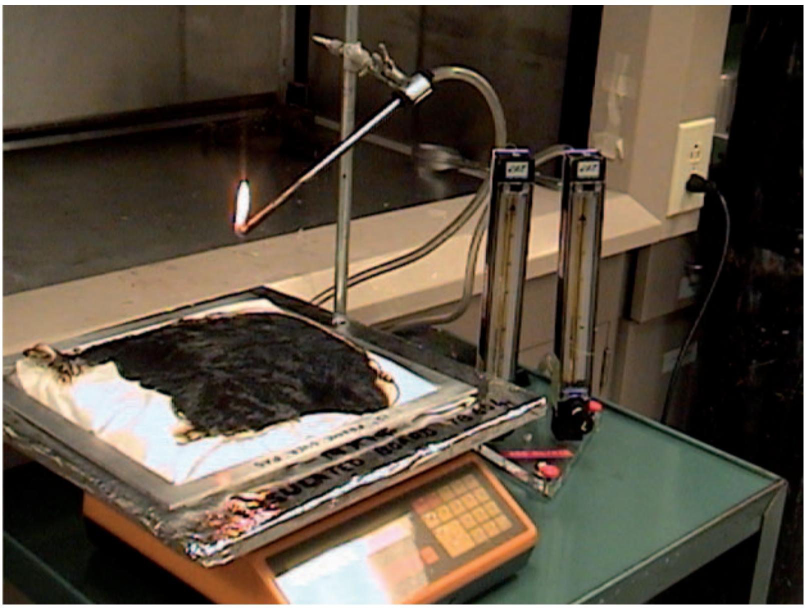

FIGURE 6. TB604 test on sample pad.

Generally LOI >28 is classified as FR. LOI of ~32 is desirable for most of the applications. This is achieved by incorporation of ~10% FR Chemical 2. Nonwovens containing a mixture of FR Chemical 1 & FR Chemical 2 did not show a good correlation with LOI values. Incorporation of nanoclay containing FR Chemical 3 in the nonwoven did not alter LOI values, but strengthened the char. In all samples, if the fiber is intrinsically FR and does not melt or burn at all, then it can function as a char-reinforcing grid.

The LOI test results relate only to the behavior of the test specimens under the test conditions in the laboratory. In real fires the conditions are

FIGURE 7. Sample pad after the TB604 Test. (Char on the fireside and change in color on other side)

entirely different and hence the LOI values must not be used to infer the fire hazards of the material. During common fires oxygen depletes, room temperature rises, and thus there is no correlation of LOI to real end use conditions; thus it is widely used as a preliminary FR screening test.

Commercial mattresses are required to pass the expensive test 16 CFR Part 1633' Open Flame Standard for Mattresses. Small industries and researchers cannot afford this test. Hence for our further research, we are using the test described by California Technical Bulletin 604 (TB604) [20] or 16 CFR Part 1634 (set up shown in Figure 6), a laboratory scale flammability test for samples of filling materials of bedding accessories (comforters, pillows, and mattress pads etc.). In this test, a sample pillow is ignited with a small open flame and allowed to burn for over 6 min. The specimen passes the test if weight loss does not exceed 25.0% and there is no flash over. A barrier pad sample passes the test if the flame does not create a void of more than 50 mm in diameter. FR properties of the barrier pads produced exceed the specific requirements of TB604. After the test the entire char formed on the sample pad (shown in the Figure 7) was intact with no holes created by the fire (not even a pinhole). Other side of the sample did not char and had slight change in color. This demonstrates that the product of this research would be a suitable candidate for bedding products to comply with the latest open flame standard.

CONCLUSIONS

By blending cotton with FR fibers (35%) the LOI value increases from 19 for cotton to 22. FR Chemical treatment enhances the flame retardancy by increasing LOI value up to 38. Out of two chemicals tried, FR Chemical 2 performance is found to be better than that of FR Chemical 1. Incorporation of FR Chemical 2 to a level of ~10% increases LOI value to 32, which should be sufficient for most of the applications. LOI tests are preliminary tests, and do not reflect real fire hazards. Laboratory scale flammability test TB604 or 16 CFR Part 1634, which is designed for the bedding products (comforters, pillows, and mattress pads etc.), was used. FR properties of the barrier pads produced exceed the specific requirements of TB604. After the test the entire char formed on the sample pad was intact with no holes created by the fire (not even a pinhole). Other side of the sample did not char and had slight change in color. This demonstrates that the product of this research could be tailored precisely for sleep products (such as pillows, mattress pads, or barrier material in mattress sets) such that they comply with the latest open flame standard.

The SEM pictures show that chemical binders assist FR chemicals to adhere onto the fibers, present even after the burning that strengthens the char. The EDS confirms presence of applied chemicals in the web. In all samples if the fiber is intrinsically FR and does not melt or burn at all, then it can act as a char-reinforcing grid. Probably the combinations of flame retarded char-forming fibers and chemicals along with nanoclay give rise to a consolidated fiber-char-reinforced structure, which enhances heat and flame resistance compared to individual chars. These interactive combinations impart high FR properties to the soft furnishings with the performance that exceeds TB604 requirements.

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BIOGRAPHY

Manjeshwar G. Kamath has a MS in Polymers (2004), BS in Chemical Engineering (1978), and is a Licensed Professional Engineer (since 2000). He has more than 25 years of industrial experience in developing extrusion grade synthetic polymers, specialty fibers, filament yarn, film, bottles, bicomponent fiber, flame retardant fiber, low melt binders, and cotton-based nonwoven pads for mattresses. He has published about 5 book chapters, 5 posters and 16 papers on fibers, polymers, and nonwovens. Presently employed at Ticona Polymers, Shelby, North Carolina; and also enrolled in PhD program at the University of Tennessee, Knoxville, graduating in Spring 2009.